

# SCIENCE FOR CERAMIC PRODUCTION

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## POROUS FILTERING CERAMICS WITH ENGOBED PORES

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The authors described the results of modernizing ceramic filters made of chamotte-bentonite ceramics through decreasing the pore size of the main carrier by means of introducing engobe into pores in the form of a multiphase solid solution of anorthite and leucite, which after secondary firing constitutes molecular sieves with pores 1.5–2.0 μm in diameter and ensures effective filtration of suspensions.

Processes in the chemical and chemicometallurgical industries widely use the method of separation of heterogeneous solid-liquid systems by filtering suspensions, in order to obtain precipitate (sludge) containing nickel, copper, molybdenum, metal oxides, as well as gold, platinum, and other materials from solutions (pulp) of various chemical reactants

Filtering plants with ceramic filters are used in technological processes for recovery of valuable components from waste gases, for biological and mechanical purification of industrial sewage, purification of liquids in food and pharmaceutical industries, and for drinking water purification.

Membrane separation of liquid and gas mixtures, in which selectively permeable solid membranes are used as filtering elements, has become common in the past decade. The output and efficiency of the technological purification processes primarily depend on the quality of highly selective permeable membranes.

The most common method for making ceramic membranes is deposition of a thin ceramic membrane film consisting of aluminum and zirconium oxides, silicon carbides, cordierite, and other materials onto a porous substrate (carrier) made of ceramics and having an isotropic or anisotropic structure [1].

The thin selective layer (membrane) deposited on a porous substrate imparts increased strength to the filtering element and ensures its service reliability.

The carrier element of the filter after sintering should have high open porosity and permeability, ensure a homogeneous distribution of pores of a preset size over the filtering block volume, contribute to decreasing hydraulic resistance to the entering flow, and increase the efficiency of filtration and regeneration [2].

Wide application of membrane filtering elements in the industry is impeded by their high manufacturing cost, which is due to technological difficulties of producing pores of a preset size with a uniform distribution over the filtering block surface.

The traditional method of suspension filtration using natural and synthetic filtering tissues and ceramic filtering elements does not guarantee an effective degree of filtration and is not cost-effective.

At present, the plants for the separation of suspensions of a fast-precipitating solid phase widely use cylindrical chamotte-bentonite cartridge filters developed by the UkrNIKhimmash Institute and manufactured by the Ekosintez Company (former Slavyansk Ceramic Works) according to TU 21-55-97 and TU-28-45-83. Such filters have some disadvantages: a wide mean arithmetic interval of pore sizes, low mechanical strength, large losses of product in the filtrate, insufficient efficiency of the plant.

We made an attempt to increase the quality parameters of industrial filters with simultaneous extension of their application areas.

The main purpose was to modify the filter skeleton by decreasing as much as possible the maximum pore size by fractionation of chamotte and improving the homogeneity of pores (bringing their post-sintering size closer to the mean arithmetic pore radius), thus increasing the mechanical strength and chemical resistance of the filter. In the second phase of the development the obtained filtering skeleton was immersed to a preset depth into a disperse slip system (engobe), and after a second sintering a membrane layer with an increased specific surface area ensuring highly efficient filtration was obtained.

The carrier skeleton of the filter was porous chamotte-bentonite ceramics ShbBK No. 21 (Table 1) with a mean hy-

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**TABLE 1**

Parameters*	Ceramic skeleton (substrate)		Modernized filters with engobe density, g/cm <sup>3</sup>		
	initial ShBK No. 21	experimental MK-15	1.46 (sample I)	1.53 (sample II)	1.56 (sample III)
Permeable porosity, %	36	40	35	32	30
Water permeability, μm <sup>3</sup>	12.0 – 25.0	16.0 – 18.0	7.5	5.6	4.2
Pore diameter, μm:					
mean hydraulic	55.0	35.0	10.0	5.0	2.0 – 2.5
main	50.0 – 65.0	25.0 – 35.0	3.8 – 4.8	2.5 – 3.0	1.5 – 2.0
Strength, MPa:					
compressive	27.5	28.5	32.0	34.0	35.5
in rupture by inner hydraulic pressure	2.5	2.6	2.8	3.0	3.2
Acid resistance, %	97.0	97.0	97.0	97.0	97.5

\* Alkali resistance in all cases was 90%.

draulic pore radius (the main radius) of 50 – 60 μm, apparent porosity 36%, water permeability 12 – 15%, and compression strength 27.5 MPa.

The initial composition of the molding mixture was as follows: (here and elsewhere in wt.%): 75 – 78 highly fired chamotte of a certain granulometric composition, 22 – 25 bentonite (montmorillonite high-plasticity clay), 5 – 8 (above 100%) water.

The skeleton samples were made in the form of hollow cylinders of size 120 × 70 mm and 250 mm high made by isostatic molding, and also flat plates 300 × 300 mm and 35 mm thick made by hydraulic molding in a metal mold [3].

After molding and drying, the molded articles were fired in a tunnel furnace at a temperature of 1250°C. Samples were cut out of fired articles and tested according to the standard methods, as a consequence of which composition MK-15 was chosen (Table 1).

These samples with specific mass and granulometric chamotte compositions were used to produce a porous skeleton and make filtering elements with engobed pores playing the role of the main filtering layer.

Natural materials of the following composition were used to produce a capillary filtering layer (engobe) (%): 28 – 35 highly fired chamotte, 22 – 27 GK alumina, 15 – 18 Prosvanovskoe kaolin, 8 – 10 Avdeevskoe quartz sand, 15 – 22 VGO clay, 12 – 15 (above 100%) Raigorodskoe chalk. A slip suspension of finely dispersed batch was prepared by joint wet milling of the components in a ball mill to a specific surface area with main particle size ≤ 0.015 mm.

Engobe was introduced into the pores of the fired filter skeleton to a preset depth by impregnation or pulverization. The required pore sizes was controlled by the degree of dispersion of milled slip suspension and its density.

To analyze the physicotechnical parameters of the engobe, samples were produced by casting in a gypsum mold with subsequent drying and firing at a temperature of 1230 – 1250°C, after which they were polished along planes.

Cut out fragments of modernized filters were used to make disks 50 mm in diameter and 5 mm thick to identify the main physicochemical parameters of samples I – III (Table 1).

The service parameters of experimental filter samples depending on the degree of pulp dispersion were identified according to well-known methods on a vacuum plant with rarefaction of 0.85 – 0.90 kPa. The parameters controlled included the volume of the filtrate obtained, the rate of sludge precipitation, the minimum amount of the retaining fraction, the content of solid material in the filtrate and the degree of its extraction, as well as the purity (clarity) of liquids (based on fuchsine).

The filtering suspension was milled feldspar with a 50% content of particles ≤ 2.5 μm.

Based on the obtained statistical data, the most efficient compositions of mixtures for the main filter substrate and the parameters of engobe ensuring effective filtration according to present parameters were determined.

At the same time, an integrated study of the chemical composition of engobe samples was implemented using the chemical, spectrographic, and atom-absorption methods. The chemicals composition of the samples was as follows: (%): 38.10 SiO<sub>2</sub>, 54.60 Al<sub>2</sub>O<sub>3</sub>, 0.91 Fe<sub>2</sub>O<sub>3</sub>, 0.30 TiO<sub>2</sub>, 0.18 MgO, 2.38 CaO, 0.52 BaO, 0.23 Li<sub>2</sub>O, 0.07 Cr<sub>2</sub>O<sub>3</sub>, 1.20 Na<sub>2</sub>O, 1.80 K<sub>2</sub>O, and 1.61 RO/R<sub>2</sub>O<sub>3</sub>.

The x-ray phase analysis of the phase composition has established that the sample has several phases; it represents a solid solution of anorthite CaAl<sub>2</sub>Si<sub>2</sub>O<sub>7</sub> and a mixture of analogs (Na, Li) of leucite KAlSi<sub>3</sub>O<sub>8</sub> with inclusions of α-Al<sub>2</sub>O<sub>3</sub> and α-SiO<sub>2</sub>. Compounds of this type are typical zeolites and constitute molecular filters ensuring highly efficient purification of liquid suspensions.

Such filters can fulfill the function of a mechanical filter, an absorbent, a molecular sieve, or an ion-exchange material and ensure high-efficiency water purification from ions of heavy metals, electrolytes, and radionuclides.

Based on the research performed, a method and a technology have been developed for producing capillary-type ceramic filtering elements for a wide range of practical applications in industrial plants for filtration of liquid suspensions and separation of neutral, acid, and alkali liquid solutions (Ukraine patent No. 39564A).

A filtering element for a disc filter has been designed to be used in industrial vacuum filters with vertical discs (Ukraine patent No. 41623A).

The filter element prototype was tested at the Noril'skii nickel JSC in an industrial plant made by the Outokumpu Company (Finland). Observations corroborated the efficiency of the filter. This prototype can be used to produce industrial products.

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